

## STANDARD OPERATING PROCEDURES FOR COATING ANNULAR DENUDERS WITH MAGNESIUM OXIDE

RTI International  
Research Triangle Park, North Carolina

Prepared by: W.C. Eaton Date: 8-15-03

Reviewed by: Jane B. Flay Date: 8-15-03

Approved by: RKM Jayaram Date: 8-15-03

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## **STANDARD OPERATING PROCEDURES FOR COATING ANNULAR DENUDERS WITH MAGNESIUM OXIDE**

### **1.0 Purpose and Applicability**

This document outlines procedures for cleaning and coating glass annular denuders with magnesium oxide for the collection of gas phase acidic species in the ambient air. This SOP is applicable for coating glass annular denuders used with the URG MASS 400 and Andersen RASS speciation samplers. Under most ambient sampling conditions, the denuder's performance will not degrade significantly over three months of continuous field use.

### **2.0 Safety Precautions**

- 2.1 Always wear latex or plastic gloves when handling solvents (ethyl alcohol or methyl alcohol) if there is a potential for spillage.
- 2.2 Always wear protective eye wear when conducting laboratory procedures specified in this SOP.
- 2.3 Read, understand, and follow the Material Safety Data Sheets (MSDS) or Chemical Safety Cards for all chemicals involved in this procedure.
- 2.3 Always keep open chemical containers in fume hoods and wear adequate protective clothing according to the MSDS sheets for that chemical. Wear dust mask when transferring dry MgO powder during weighing operations.
- 2.5 Always label secondary containers used in this procedure.
- 2.6 Work in a laboratory hood when transferring alcohol, alcohol/MgO slurries, and when removing MgO powder from the denuder with laboratory tissue, cotton swabs, and/or a stream of air or nitrogen gas.

### **3.0 Equipment and Materials**

- 3.1 Plastic or latex disposable gloves, sized to fit. RTI stockroom item.
  - 3.2 Magnesium oxide, light powder, USP. 500 g. EM Science, product No. MX0064-1.
  - 3.3 Ethanol (ethyl alcohol). 500 mL. RTI stockroom item. 200 proof, absolute.
  - 3.4 Alternate slurry liquid. Methanol (methyl alcohol). 4 liter plastic bottle. J.T. Baker product 9076-03.
  - 3.5 Hydrochloric acid solution. 2 normal (2N). Prepared by 1:3 dilution of 6M reagent grade concentrated acid. Work in a hood. Wear safety glasses and rubber gloves.
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Pour 2 parts of distilled water into a glass beaker. For example, pour 40 mL of water into a 100 mL beaker. Pour 20 mL of 6N hydrochloric acid into a glass graduated cylinder. Then slowly add the 20 mL of acid to the 40 mL of water to make 60 mL of 2N acid solution.

- 3.6 Wheaton bottles, 125 mL capacity with screw caps and plastic or Teflon cap liners. Must be suitable for use with wrist-action shaker.
  - 3.7 Teflon-coated magnetic stir bar, 1-inch length, and magnetic stirrer.
  - 3.8 Funnel, glass, powder. 80 mm width, 60 degree slant. To transfer dry MgO powder and slurries of MgO.
  - 3.9 Laboratory tissues, cotton-tipped swabs, bag ties made of thin wire coated with paper (for removing misplaced MgO slurry deposits).
  - 3.10 Distilled water, laboratory grade. Available from in-house supply, RTI building No. 6.
  - 3.11 Burrell Model 75 or equivalent brand wrist-action shaker with clamp attachments to hold bottles of MgO slurry. Located in Room 104, Building No. 6.
  - 3.12 Graduated cylinder, glass, 50 or 100 mL capacity. Graduated to nearest 1 mL.
  - 3.13 Spatula, stainless steel. For transfer of magnesium oxide from reagent bottle to slurry bottle during weighing.
  - 3.14 Top-loading electronic balance. 1200 g. capacity. Readable to nearest 0.01 g. Mettler P121D. Located in Room 208, Building No. 6.
  - 3.15 Quality control test weights, 5, 50, and 500 g.
  - 3.16 Caps and/or plugs (plastic) for sealing denuder tubes during slurry application and for closing prepared denuder tubes during storage and during shipment to collection sites. Plastic foam sleeves (pipe insulation) to protect denuder tubes from breakage.
  - 3.17 Source of clean, dry air or nitrogen. Flexible Tygon tubing for routing air or nitrogen to denuder tubes during drying stages. Air and nitrogen are available at hoods in Buildings 3 and 6.
  - 3.18 Annular denuders, clean and dry, equipped with caps and/or plugs and labeled by the Sample Handling and Archival Laboratory (SHAL). If the denuders are not clean, hold each securely and rinse it from one end, then the other with a stream of warm tap water to wet the used MgO coating. Since this cleaning process does not fully remove the coating, 2N hydrochloric acid must be used. Proceed as follows, working in a hood. Drain excess water from the denuder. Cap the exit end of the denuder. Slowly add 25 mL of 2N acid to the open end (the recessed end) of the denuder. CAUTION! Gas will be evolved. Without capping the denuder, tilt it slowly so the acid solution flows towards the open end, but do not tilt so far that acid pours out. Check again for fumes and gases. When gas evolution ceases, cap the end and while holding both caps securely against the ends of the denuder, invert slowly in a back and forth motion for five repetitions. Uncap one end of the denuder and pour the acid down the drain. Rinse both the denuder and the drain opening with plenty of tap water. Remove the other cap from the denuder. Clean both caps in tap water, then distilled water to remove acid remnants and set aside to dry. Then immediately rinse the denuder interior
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and exterior with copious amounts of warm tap water, followed by multiple rinsing with a stream of distilled or deionized water from the laboratory tap, taking care to pass water through each annular opening. Finally, pass a stream of air or nitrogen through the denuder to remove most of the water. Let each denuder air dry overnight before coating it. If a denuder is needed sooner, continue passing a stream of laboratory air or nitrogen through the tubes until all surfaces are dry. Alternatively, ethanol or methanol can be added to the denuder to rinse out the water and then dry the denuder with a stream of air or nitrogen.

#### **4.0 Preparation of Magnesium Oxide Slurry**

- 4.1 Use spatula to quantitatively transfer 12.5 g of magnesium oxide into a labeled, preweighed, Wheaton bottle/powder funnel using top-loading balance. Cap the bottle. Record information in laboratory notebook. Several bottles may be prepared at the same time, depending on the need. One bottle of prepared slurry is enough to coat at least three URG or Andersen annular denuders.
  - 4.2 Working in the hood, use the graduated cylinder to add 40 mL of ethyl alcohol to the Wheaton bottle containing the MgO powder. Cap the bottle securely. Tilt the bottle and contents back and forth several times until all the powder is wet with alcohol.
  - 4.3 Attach the bottle and contents to the wrist-action shaker. Counterbalance the slurry bottle with another on the other side, or use an equivalent weight of water in a bottle to counterbalance.
  - 4.4 Set the shaker to high speed shaking, and set the time period to 15 minutes.
  - 4.5 After two minutes shaking, stop the shaker and check the tightness of the clamps holding the bottles. Tighten the clamps (if necessary) and then restart the shaker.
  - 4.6 After 15 min, remove the slurry bottle.
  - 4.7 Open the bottle and insert a Teflon-coated magnetic stirring bar. Replace the cap and place bottle on a magnetic stirrer. Stir at "low" or "2" setting for an hour. Immediately take the bottle containing the slurry to the hood where the denuders are to be coated. Shake the bottle from time to time (every 5 minutes) to prevent the slurry from settling. Begin use immediately to coat denuders.
  - 4.8 An alternate procedure to the use of the Wheaton bottle and wrist-action shaker is as follows. Place multiples of 12.5 g magnesium oxide/40 ml ethyl alcohol in a glass bottle. Add a stirring bar, cap the bottle, and shake the contents until all the magnesium oxide powder is wetted. Place the bottle on the magnetic stirrer and stir for 5 to 15 minutes. Remove the bottle and hand-shake the contents so that excessive deposits adhering to the upper part of the bottle interior is mixed into the slurry below. Put the bottle back on the magnetic stirrer and continue to stir at a low setting until ready for use. Return the bottle to the stirrer immediately after withdrawal of slurry to keep the contents well mixed and ready for the next use. If the magnesium oxide settles out, use the stainless steel spatula to dislodge the stirring bar and break up the solids. Recap
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and shake the bottle well; return to the stirrer and stir until the contents appear homogeneous and free of any visible clumps of magnesium oxide.

## 5.0 Coating of Glass Annular Denuders with Magnesium Oxide

- 5.1 Inspect each glass denuder carefully to be sure it is not broken nor detached from its points of connection to the cylinder that holds it inside the metal tube. Broken glass will be evident. Tap the denuder gently on a black piece of paper; no glass particles should fall out. One technique to examine the denuder is to hold it up to a light and sight through the glass tubes looking for signs of detachment.
  - 5.2 Initially clean each denuder with distilled or deionized water and allow to air dry. Alternatively, the denuder may be dried with a stream of dry air or nitrogen. Be sure the identifying bar code label is firmly attached. Refer to Step 3.17 above.
  - 5.3 Remove all caps or plugs and weigh the clean, dry denuder on the top-loading balance to the nearest 0.01 g. The uncoated URG downtube denuder has an internal volume of approximately 63 mL and weighs about 600 g. The uncoated Andersen denuder has an internal volume of approximately 35 mL and weighs about 460 g. Record information in the laboratory notebook dedicated to denuder preparation. Return the denuder to the hood to start the coating process.
  - 5.4 Cap the exiting end (the end with the greater distance between the glass denuder and the metal lip) of the Andersen denuder with a screw-on polyethylene cap. Plug the URG downtube denuder with the plastic fitting that is held in place by the two O-rings in the exiting end of the denuder.
  - 5.5 Shake the denuder slurry bottle again. Remove the bottle cap. With one hand, hold or prop the denuder, capped end down, perpendicular to the floor of the laboratory hood. Pour about half of the volume of slurry into the open end of the denuder. Cap the slurry bottle and put it aside. Cap the end of the denuder with the screw cap (Andersen) or a plastic fitting (URG).
  - 5.6 Invert the denuder and allow the slurry to flow to the other end. Bring the denuder tube to a lengthwise, horizontal position. Rotate the denuder tube on its lengthwise axis about 90 degrees, then tilt the denuder 45 degrees and allow the slurry to flow to the other end. Repeat this process 4 or 5 times, tilting to the left, then to the right, until the tube has been rotated back to its starting position.
  - 5.7 Place and hold the denuder perpendicular on the floor of the hood. Remove the top cap, then drain and discard the excess slurry into the slurry bottle via the funnel. Cap the slurry bottle. *Note 1: While making this transfer, the denuder needs to be slowly and continuously rotated to provide a uniform coating. If later the coating appears streaked, uneven, or is plugging large portions of the tube, clean and dry the denuder and repeat the coating process. Note 2: The ends of the denuders will have excess MgO slurry present which will dry quickly. This will be removed in a later step.*
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- 5.8 Immediately after draining the excess slurry, place the denuder tube horizontally on a layer of paper towels on the floor of the hood and gently roll the tube back and forth for approximately 5 minutes to encourage uniform coating of internal surfaces.
  - 5.9 While continuing to roll the tube, pass a slow (no more than 10 liters per minute) of air or nitrogen through one end then the other end of the denuder tube. Do this for 5 minutes.
  - 5.10 Allow the denuder to air dry overnight, or if need be, dry further with laboratory air or nitrogen.
  - 5.11 Using the stainless steel spatula, damp lab tissues, cotton swabs, remove the excessive coating from the metal and/or O-rings at each end of the denuder, making a smooth straight line where the coating ends on the glass surfaces. Hold the tube up to a strong light and rotate it to examine each of the annular spaces. If the MgO has “bridged” any of the denuder tubes near the ends, gently dislodge the material with a paper-covered twist-tie. If there are numerous “bridges” within the interior of the denuder, clean it and recoat.
  - 5.12 Turn on an air or nitrogen source, pinch the end of the delivery tube slightly to create a fast flow of gas, and blow any loose coating from the interior of the tubes. Do this in the hood. A slight puff of MgO powder may be observed at the exiting end but should no reappear when the gas is blown through the tube again. Turn the tube around and blow gas through the other end.
  - 5.13 Tap the ends of the coated denuder gently against a black surface to test for any residual loose MgO. Clean and recoat any denuders which show the presence of any uneven or loose coating.
  - 5.14 Reweigh the denuder on the top-loading balance. Record the value in the laboratory notebook and calculate the amount of MgO deposited. Compare the weight deposited to results from previous coatings of other Andersen or URG denuders to check for uniformity of the coating process.
  - 5.15 Cap both ends of the denuder, ensure the bar code label is still firmly attached, and return it to the SHAL for storage or shipment to the field.
  - 5.16 Remove the stirring bar from the slurry bottle with a magnetized stirring bar retriever. Clean the stirring bar and store for the next use. Recap the slurry bottle for later use or discard contents and clean bottle for the next use.
  - 5.17 If a very large volume of slurry remains, label the bottle with the date of preparation and the contents, leave the stirring bar in the bottle, cap the bottle tightly to avoid loss of alcohol, and store it on the shelf in the laboratory. At the next use, use a stainless steel spatula to dislodge the stirring bar and break up the solidified magnesium oxide. Cap the bottle, place on the magnetic stirrer, and stir until a homogenous slurry is obtained.
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